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UNIVERSITY OF TECHNOLOGY SYDNEY

CENTRE FOR **FORENSIC SCIENCE**

The Detection of Organic Explosives using Lab-On-A-Chip Technology and Related Research

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PROJECT BACKGROUND

- NIFS Innovation Strategy Pilot Project
    - *"Improved Monitoring and Detection Capabilities To Minimise the Potential Criminal Use of Explosives in Australia"*
    - Evaluated currently available instruments
    - Identified emerging technologies
        - **Lab-on-a-chip (LOC)**
    - LOC feasibility study to demonstrate the detection capability with organic explosives

PROJECT BACKGROUND

- National Security Science & Technology Unit (NSST) Project
  - *"Development of field portable devices for the detection of explosive residues"*
  - Commenced in June 2006
  - Optimise lab-on-a-chip method for analysis of organic explosives
  - Design of a custom designed device for explosives detection

LAB-ON-A-CHIP TECHNOLOGY

- Utilises micro-fabrication technology to miniaturise conventional laboratory processes

- Integrates multiple laboratory processes onto a chip
  - Injection
  - Separation
  - Detection

- Rapidly evolving area of research

LAB-ON-A-CHIP TECHNOLOGY

- Advantages of LOC technology:
  - Increased automation
  - Rapid analysis time
  - Minimal reagent consumption (μL)
  - Reduced costs
  - Potential for portability

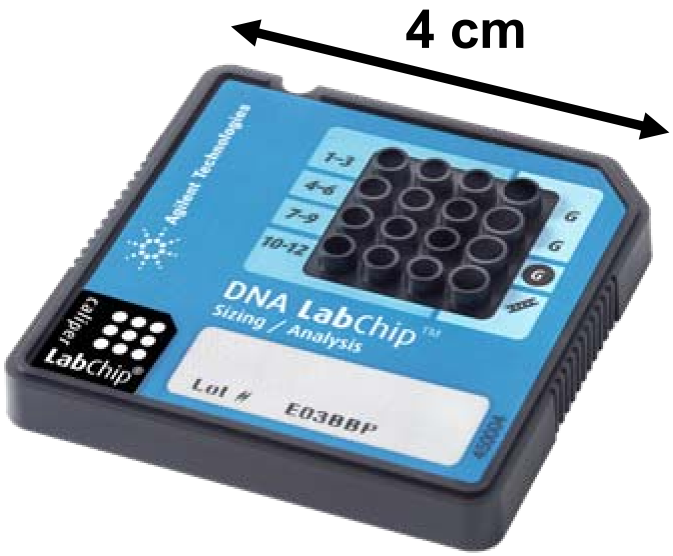
AGILENT 2100 BIOANALYSER

- One of the first commercially available instruments to employ LOC technology

- The “2100 Bioanalyser” was designed for life science applications:
  - DNA analysis
  - RNA analysis
  - Protein sizing analysis
  - Flow cytometry

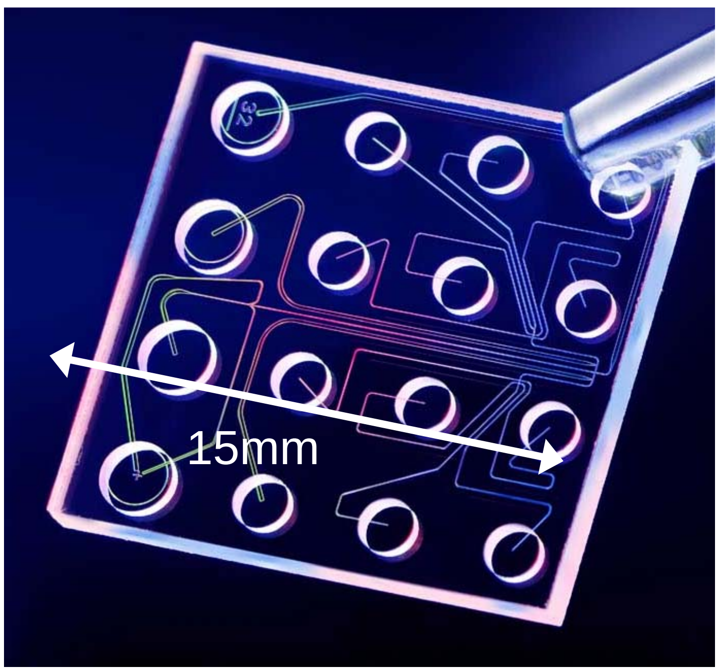
BIOANALYSER CHIP

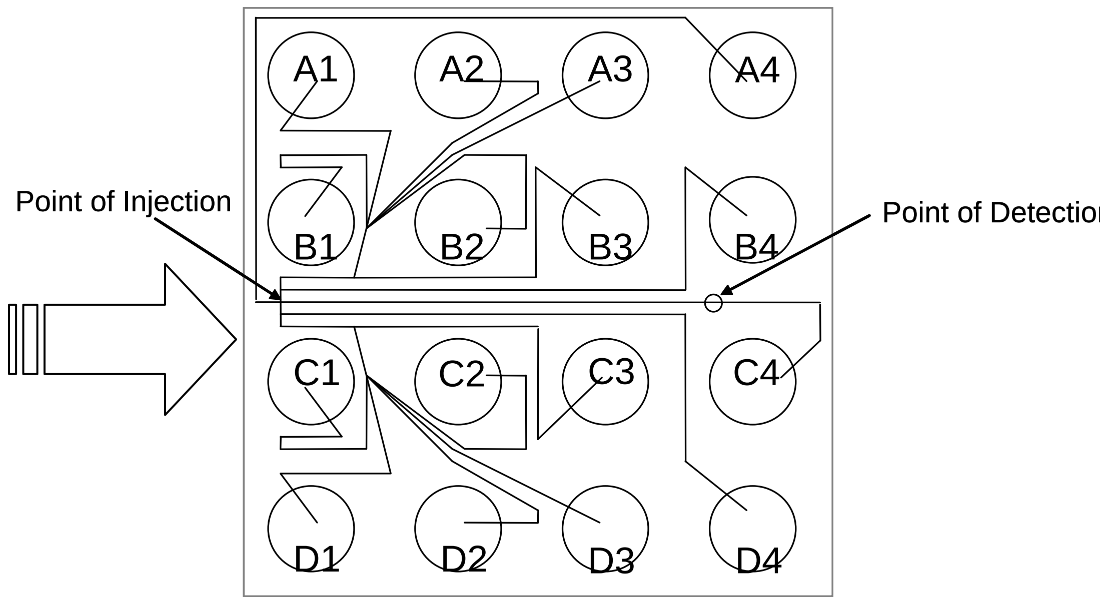
- Manufactured by Caliper™
  - Borate silica glass chip
  - 12 sample wells per chip
  - Disposable



- The ‘chips’ contain a network of interconnected channels and reservoirs

BIOANALYSER CHIP ARCHITECTURE





CHIP PREPARATION

- Samples and/or standards and electrolyte are added to wells

ANALYSIS

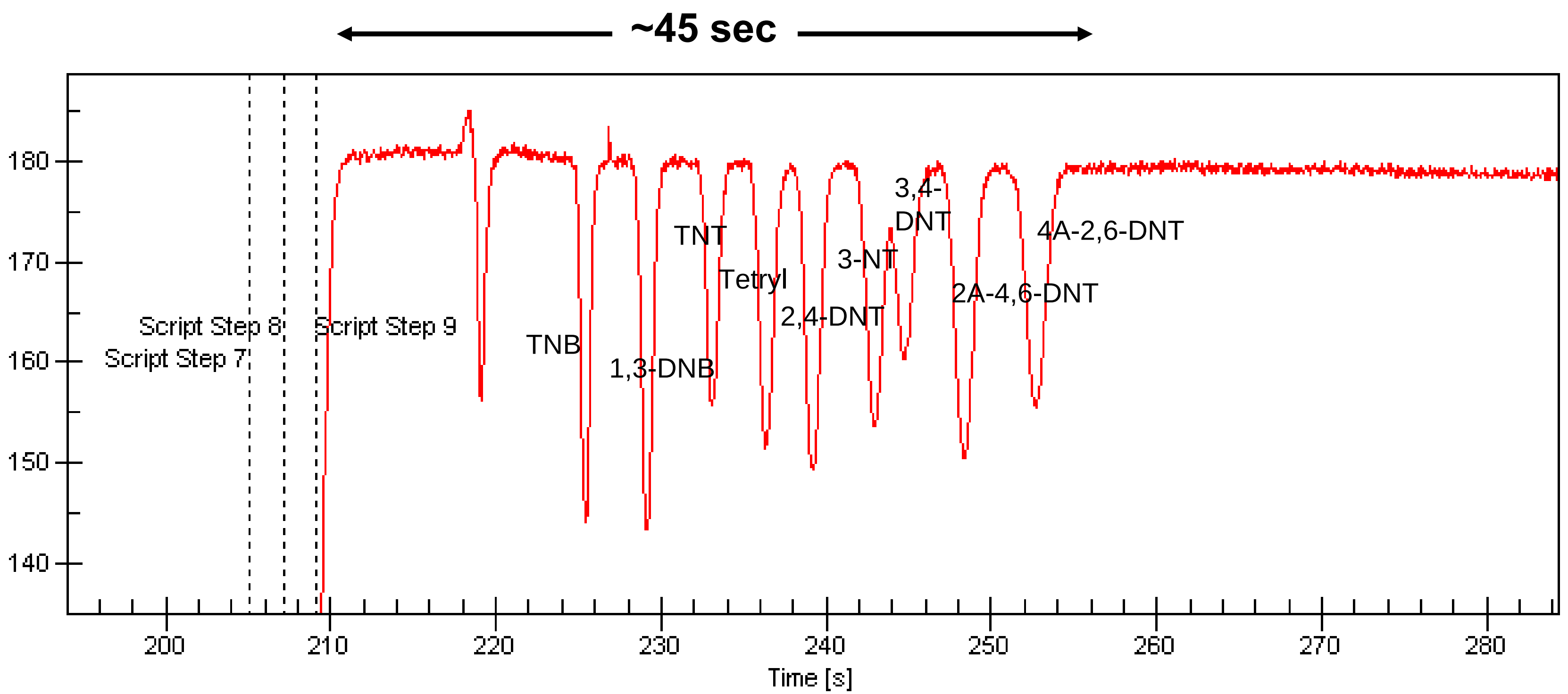
- Prepared chip is placed in the Bioanalyser for automated analysis to commence.

- Movement of solutions around chip controlled through “scripts”

DETECTION SYSTEM

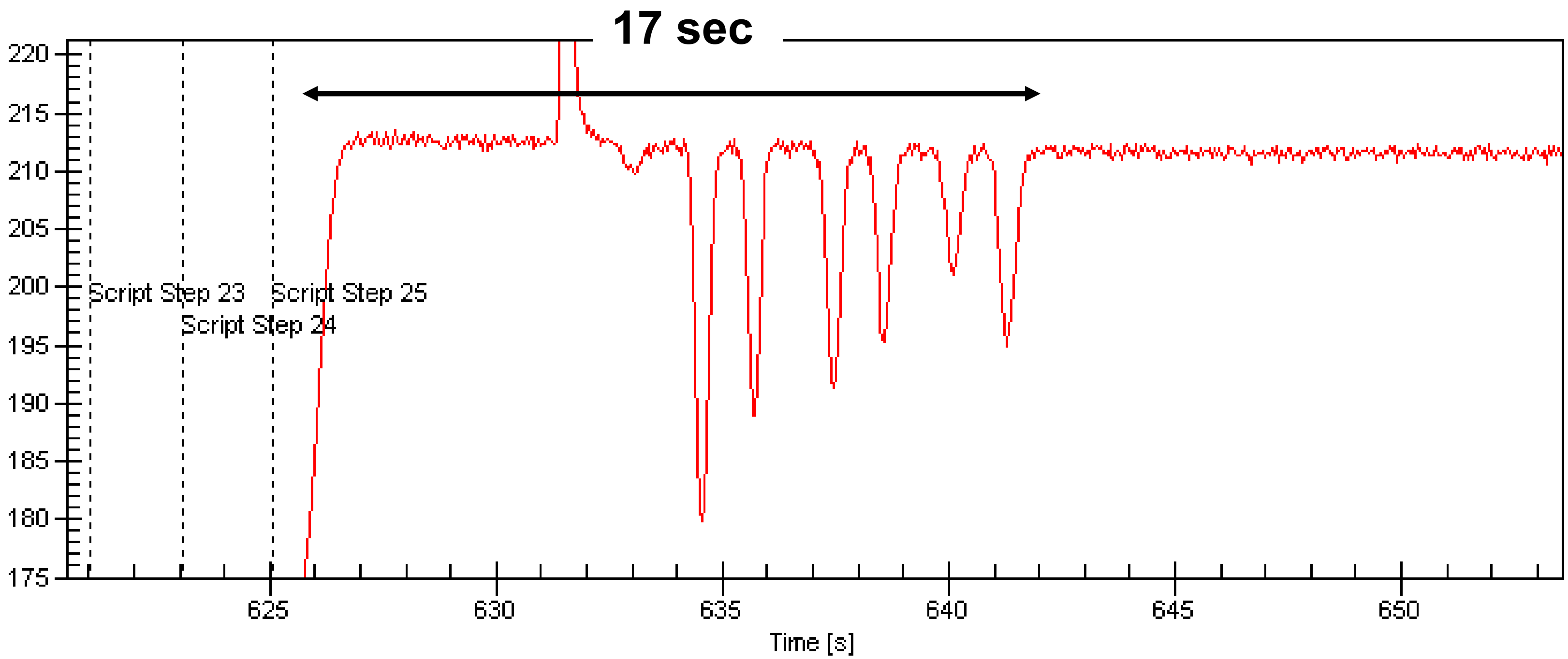
- Organic explosives are known to quench fluorescence:
  - Detection can be achieved by addition of a suitable fluorescent dye to the electrolyte
  - Explosives are detected by a decrease in the fluorescence signal
    - Indirect fluorescence detection
    - *Negative peaks*

OPTIMISED SEPARATION



- Indirect fluorescence detection

REDUCED ANALYSIS TIME



- Reduced SDS concentration (25mM)
- Fewer explosives separated

LIMIT OF DETECTION

	TNB	TNT	3,4-DNT
Limit of Detection *(ppm)*	0.15	0.25	0.50
Minimal Detectable Mass *(femtogram - x 10^{-15}g)*	3.75	6.25	12.5

- Minimum detectable mass based on estimated injection volume of 25pL
- Minimum sample volume = 6μL (TNT 1.5ng)

DETECTION OF NITRAMINES

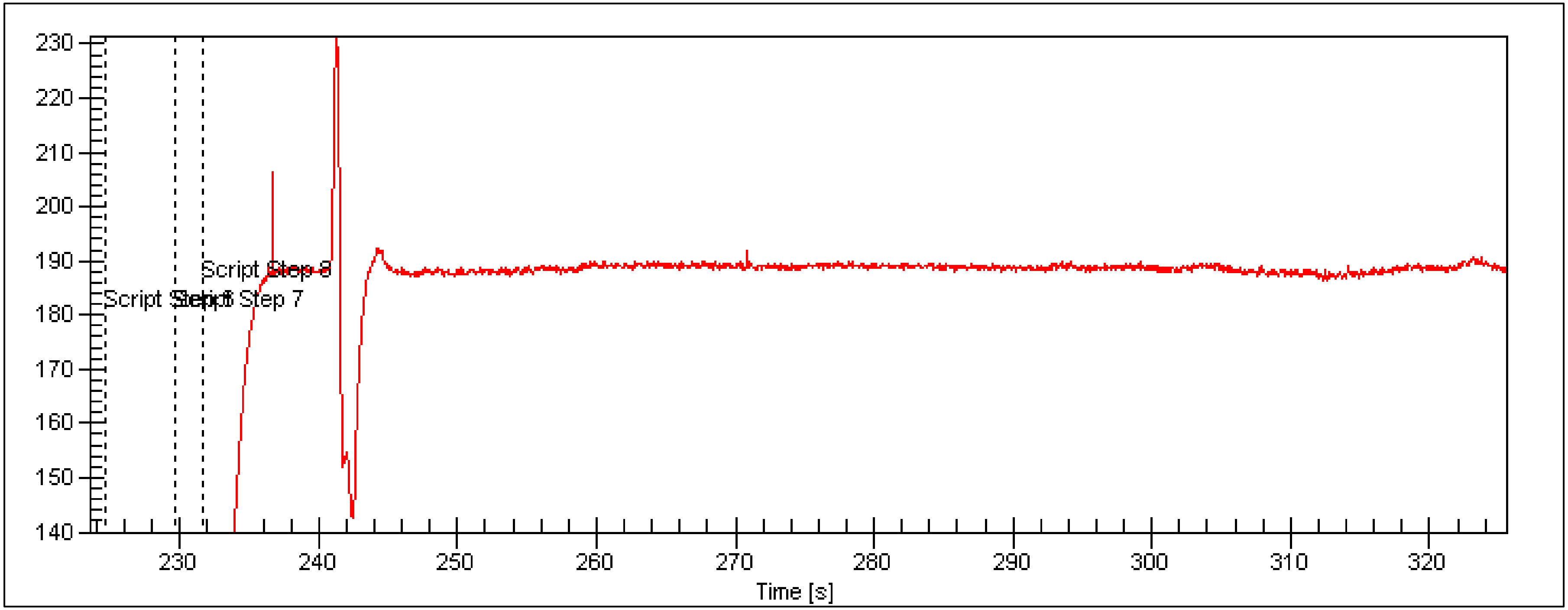
- Unable to detect RDX and HMX
  - Co-migrating with EOF
    - Increasing SDS concentration
    - Investigation of β-cyclodextrins
  - Poor quenching efficiency
    - Investigation of alternative dyes

ON-CHANNEL DETECTION

- Surface bound fluorophore (pyrene) quenching by organic explosives
  - Covalently bound to silica particles during feasibility trial

- Explosives successfully detected
  - Problems associated with stability of bound fluorophore
  - Inferior LOD

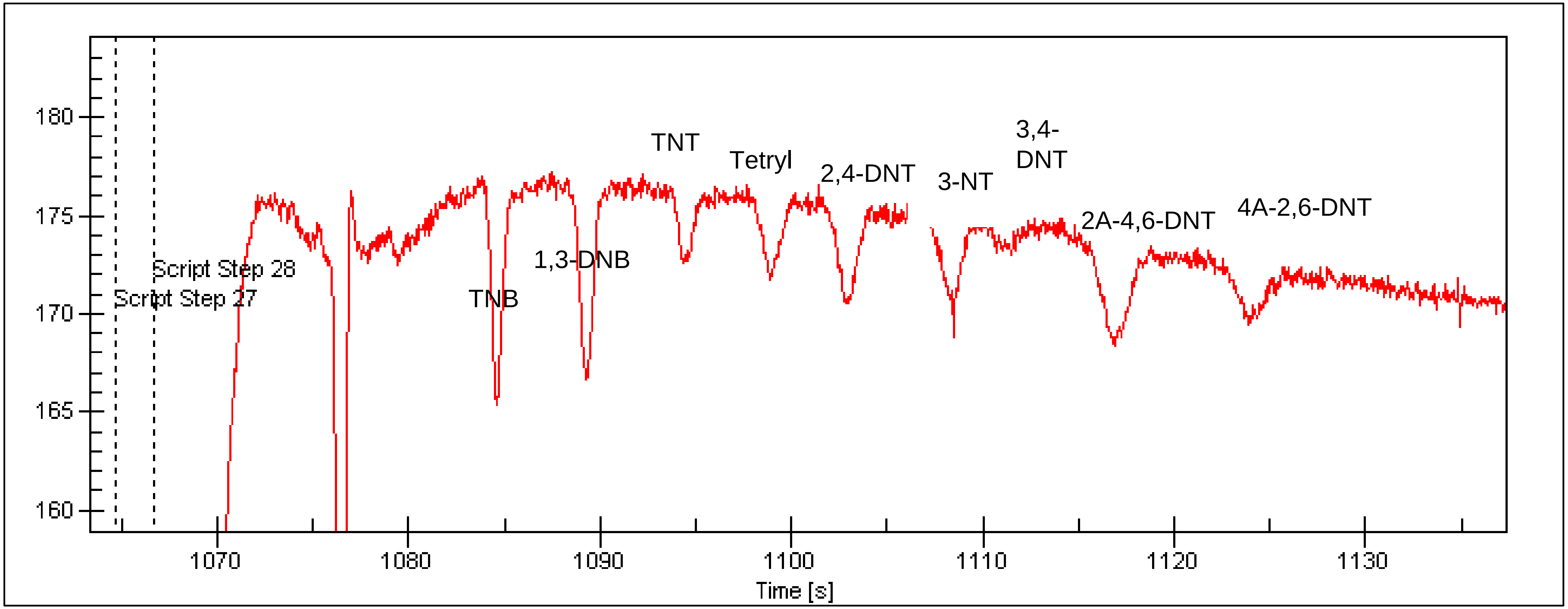
BLANK SOIL SAMPLE



- Clean, no interfering peaks

SOIL EXTRACT

5ng of each explosive



POST-BLAST SAMPLES

Generating post-blast residues for method testing

POST-BLAST SAMPLES

300g TNT Device

Capture plate

POST-BLAST SAMPLES

Generating post-blast residues for method testing

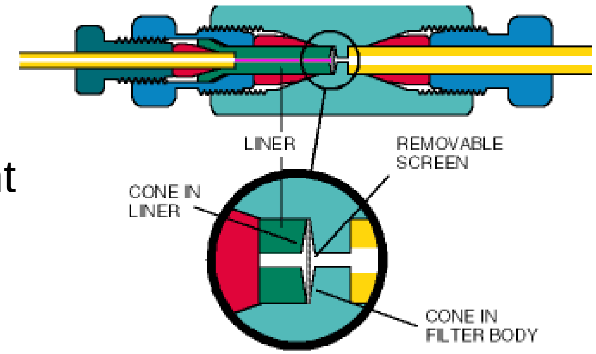
SAMPLING PROTOCOL

- Optimisation for post-blast residue sampling protocols
  - Swabbing methods
  - Soil extraction
- Low minimal detectable concentration (femtogram)
  - High concentration (ppm) LOD
- Sample pre-concentration
  - SPE
  - micro-SPE

micro-SPE

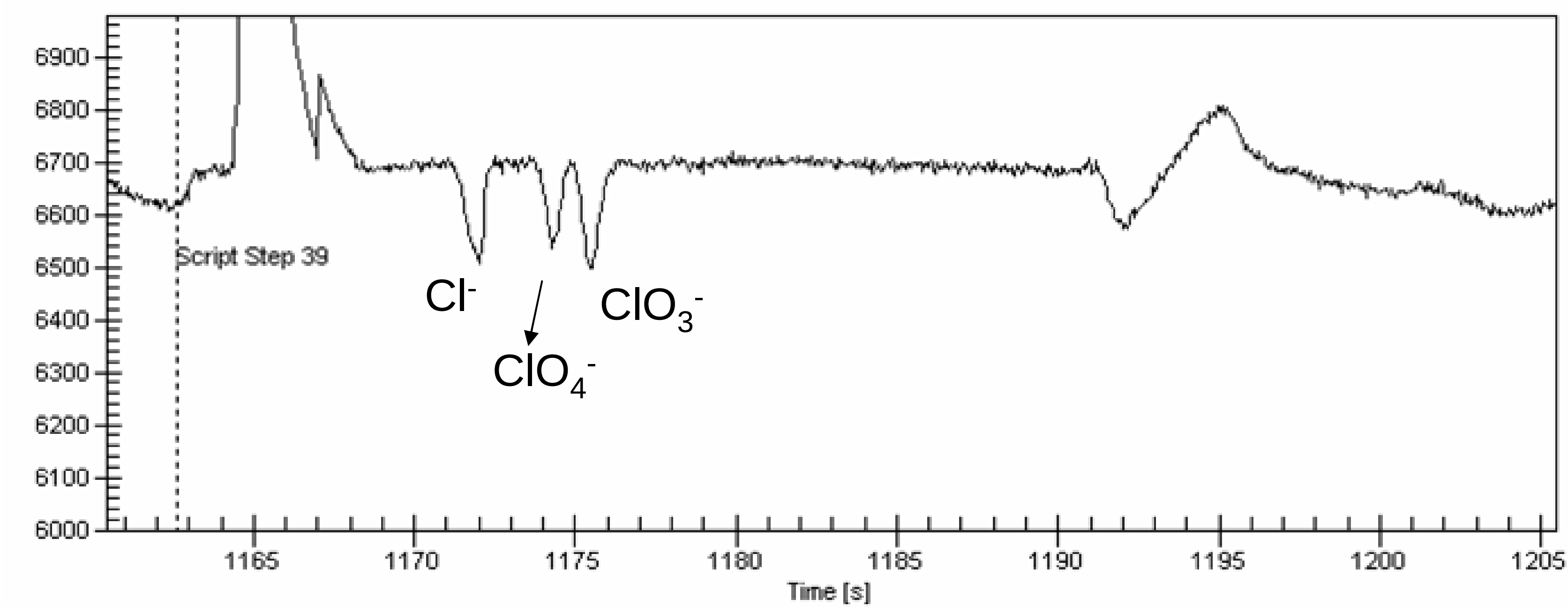
- Standard SPE cartridges
  - Large volumes of organic solvents required

- Investigating use of micro-SPE
  - Requires small volumes (<20μL) of organic solvent
  - No need for evaporation
  - Improvement in LOD
    - 25 fold decrease for TNT
    - 250ppb→10ppb



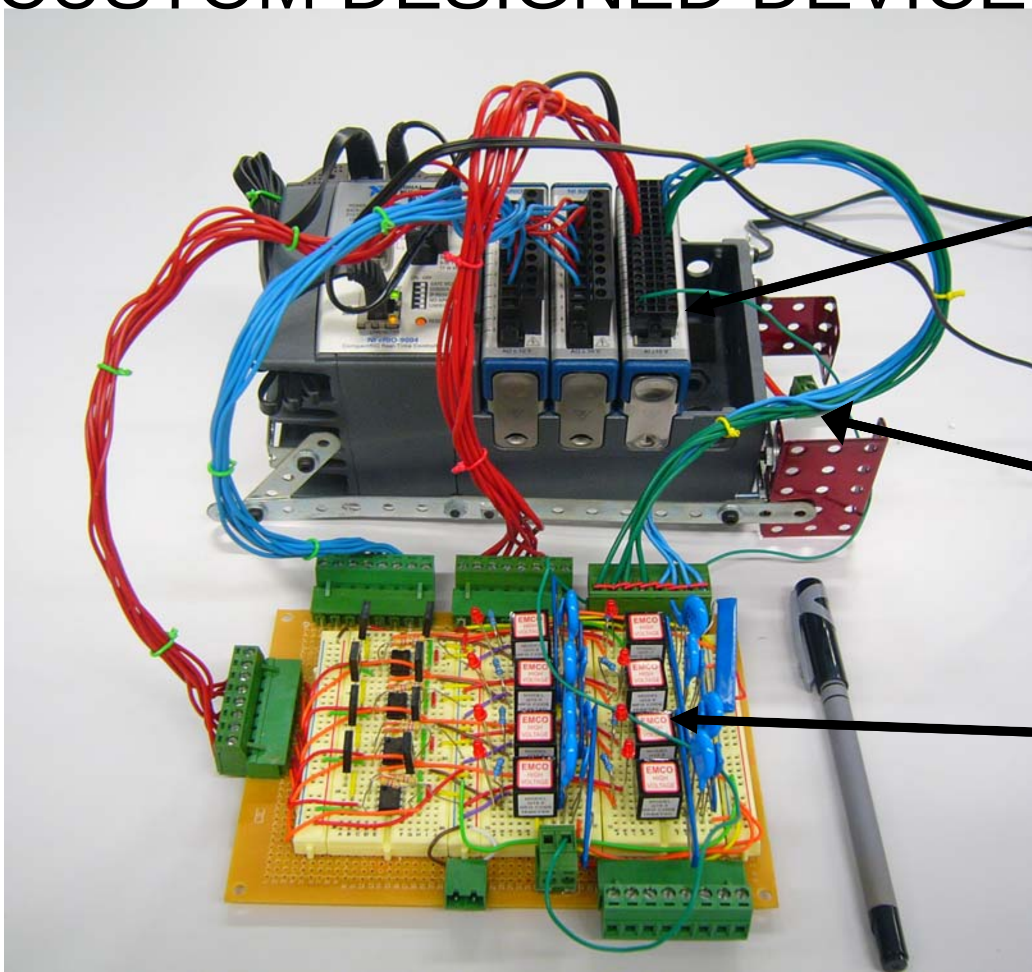
1/8" to 1/16" reducing filter with a removable screen (ZRUFR21)

INORGANIC EXPLOSIVES



- 25ppm sample in 0.025mM CTAOH, 1mM sodium tetraborate, 1mM HPTS (pH 9.3).
- Separation in 15 sec

CUSTOM DESIGNED DEVICE



National Instruments

Compact RIO Controller

Ocean Optics

USB4000 Spectrometer

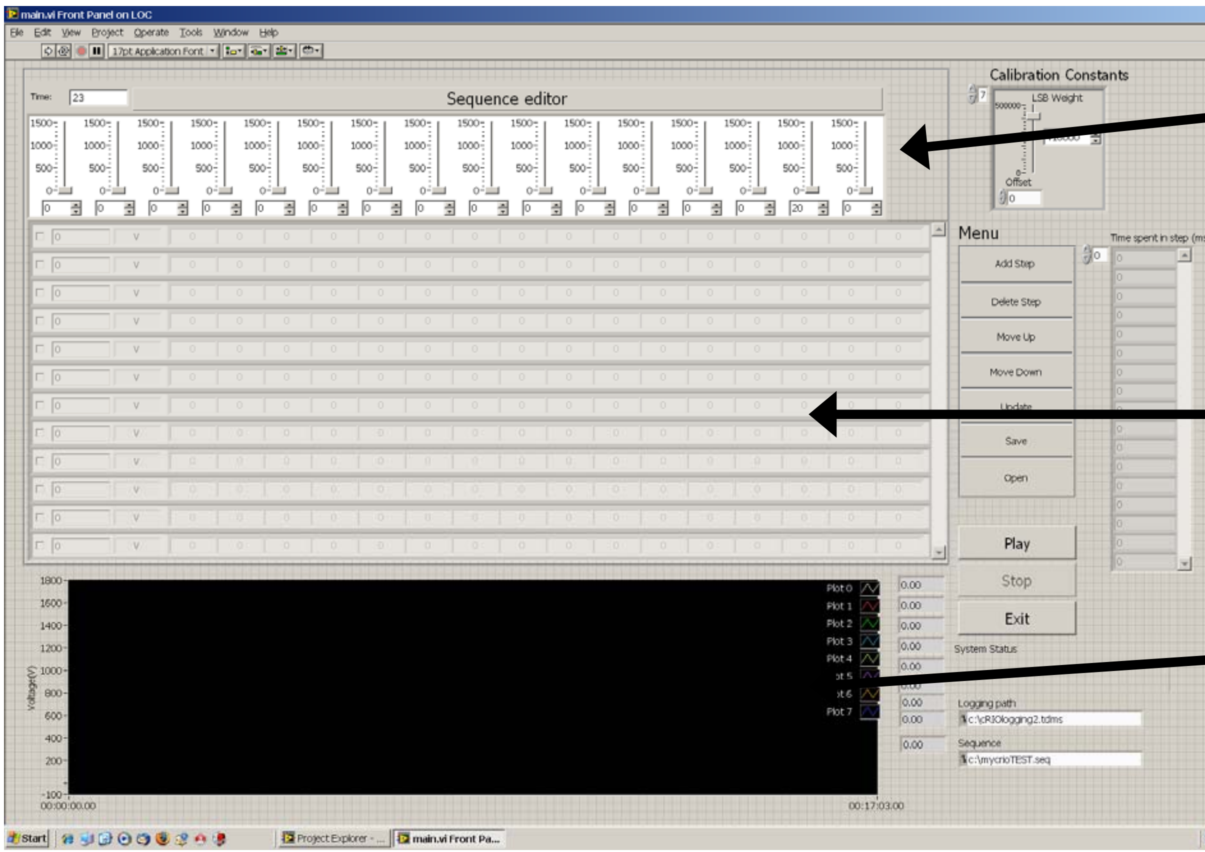
Custom Designed Circuit Board

DETECTION

Ocean Optics
USB4000 Spectrometer

- Weight 190 g
- 200-850nm

LABVIEW^{TM} SOFTWARE



Voltage control sequence for each electrode

Sample loading

Injection

Analysis

Review of voltage sequence

Voltage output for each electrode

CONCLUSIONS

- The Agilent 2100 Bioanalyser shows potential as a portable instrument for the detection of organic and potentially inorganic explosives

- Detection limits of sub ppm
- Rapid separation (~45 secs)
- Further miniaturisation currently in development

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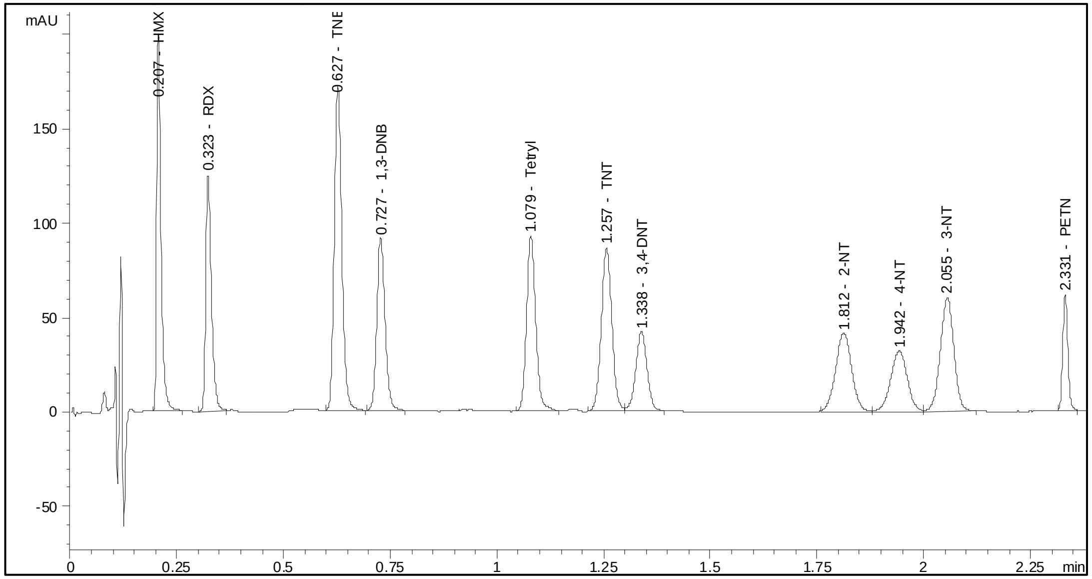
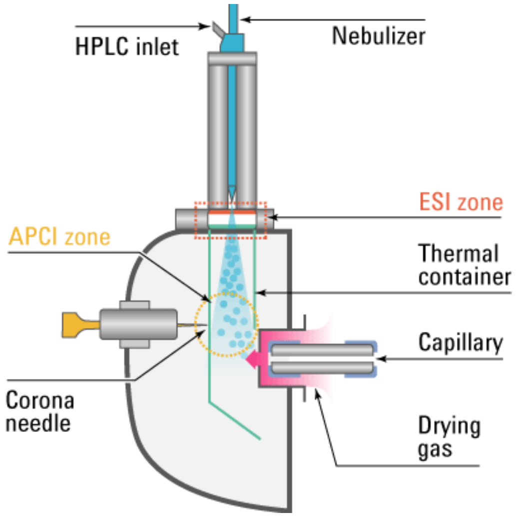
Analysis of Organic Explosives by Rapid Resolution LC

- Successful development of a rapid resolution HPLC method
  - 11 organic explosives separated within 2.3 minutes
  - Minimal detectable mass range: 110-580 pg (using UV detection)

- EPA 8330 method
  - 24 min run time



Explosives Detection via LC -Triple Quadrupole Mass Spectrometry (LC-QqQ MS)



- Agilent 6410 Triple Quadrupole Mass Spectrometer
  - Multi-mode ionisation source
    - Simultaneous ES and APCI
- Superior detection technique to the current EPA 8330 method:
  - Significant improvements in sensitivity
    - Minimal detectable mass range: 7-58 pg
    - EPA Method: 2-1400 pg
  - Rapid analysis when coupled to rapid resolution LC method
    - 2.3 minutes run time

Characterisation of Ammonium Nitrate Using Isotope Ratio Mass Spectrometry – Sarah Benson's PhD

- **Nitrogen - AN**
  - AN samples from Australia and some from SE Asia obtained
  - Bulk N method/procedures validated for AN and other inorganics
  - Cannot differentiate AN samples from Australian manufacturers using N alone
  - Significant variation between pre and post blast N in AN
- **Oxygen & Hydrogen - AN**
  - Lab stds for AN selected and under evaluation
- **Carbon – TATP**
  - Bulk C isotope ratio appears to vary based on manufacturing procedure
- **Database -** template developed

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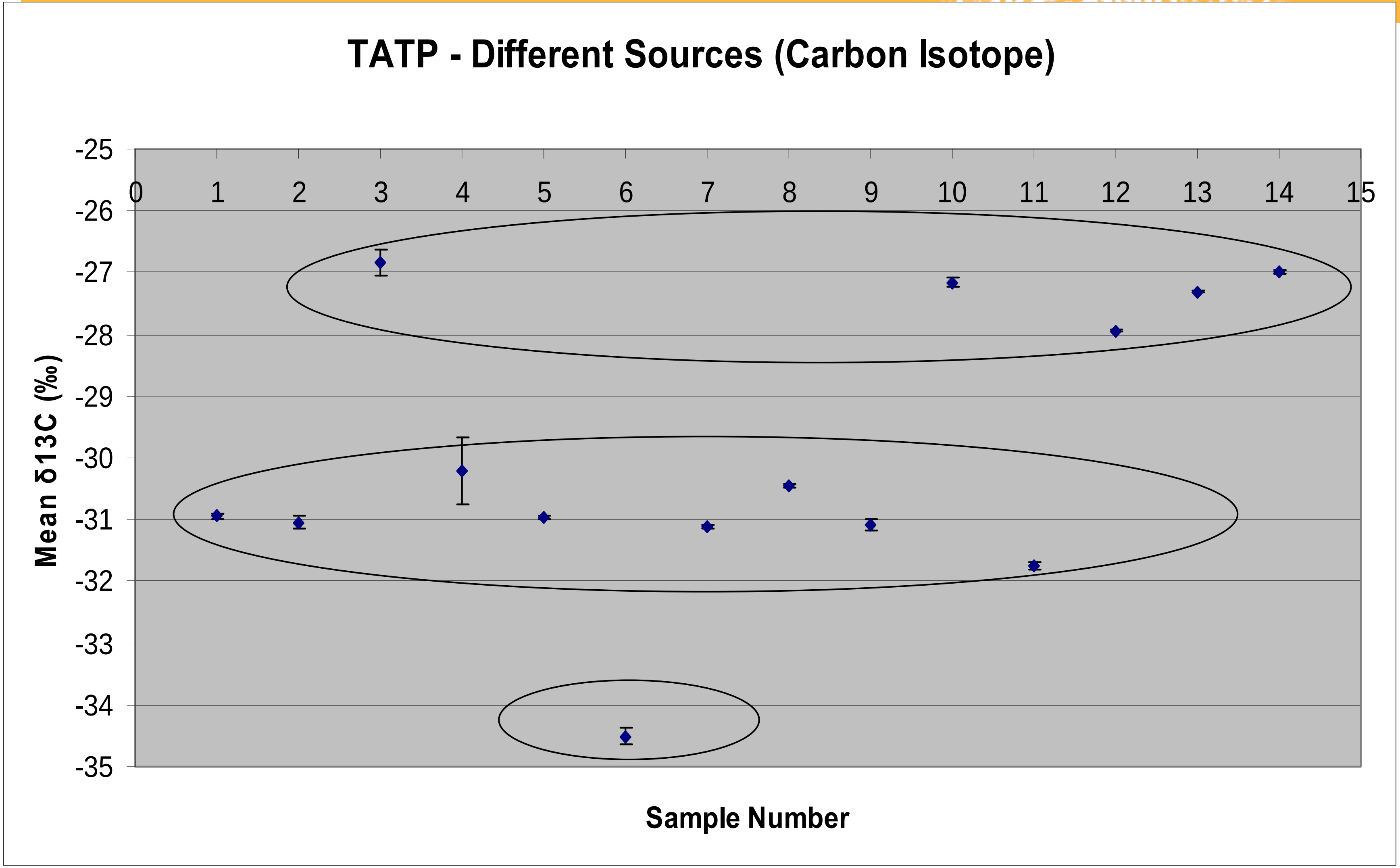

UNIVERSITY RESEARCH - Uni of Canberra, Nopporn Song-im's PhD

- Explosive residue analysis: evaluation of sampling, storage and clean-up protocols
  - TNT, RDX, PETN, TATP, Chlorates, AN
  - Outcomes
    - a universal sampling procedure for the collection of both organic and inorganic explosives residues
    - recommendations for storage of swab and extract (condition and container)
    - a better understanding of sample breakdown and the maximum period of time that explosive traces can still be detected on various surfaces.

Back to Basics for Intelligence and Investigation Purposes

- Flash Powder Mixture found at Presumed Bomb Making Facility
- Mixture of potassium chlorate, aluminium powder, and sulfur

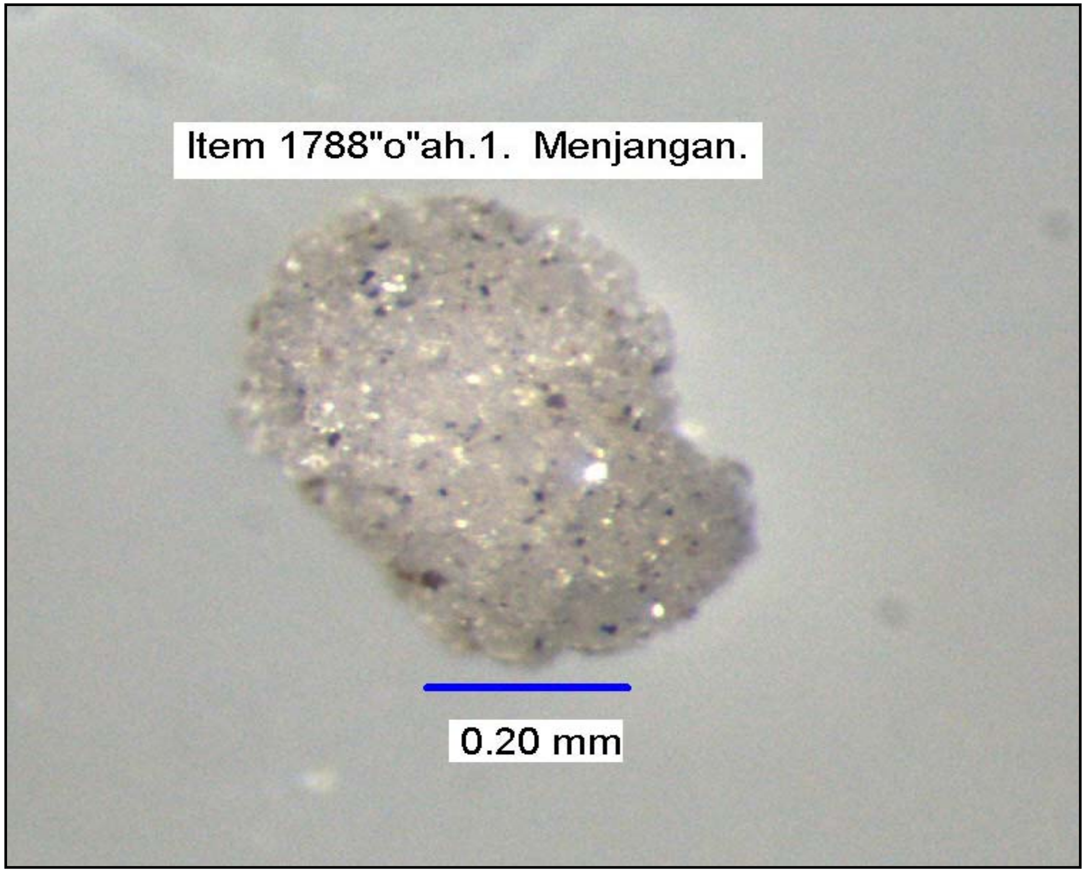

Photo: David Roylds, Australian Federal Police

ACKNOWLEDGMENTS

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- National Institute of Forensic Science
- Australian Bomb Data Centre

Australian Government

Department of the Prime Minister and Cabinet